

**Remediation  
Sampling Plan  
ESE Alcohol Inc.  
Leoti, KS**

**I. Introduction**

ESE Alcohol, Inc. (“ESE Alcohol”), on behalf of itself and Pioneer Hi-Bred International, Inc., has developed this sampling plan strictly for remediation via crop rotation per the request of USEPA-7 in order to determine the residual concentration of the seed treatments which were applied to the feedstock grain utilized to produce ethanol. EPA has identified the potential for some residual amounts of seed treatment to be present in the agricultural fields where the conditioned mash from the ethanol production was applied for the agronomic value as nitrogen fertilizer and soil conditioner. This will be a tiered sampling plan in relation to the ESE ethanol production facility located 310 East Highway 96, Leoti, Kansas (the “ESE Alcohol Facility”), and nearby agricultural fields where byproducts from the ESE Facility were land applied as fertilizer/soil conditioner and irrigation water since January 1, 2020 (the “Site”). The tiered approach shall afford the refining of the sampling process as higher application rate fields are sampled first.

The ESE Alcohol facility located in Leoti, Kansas utilized discard treated seed, as their primary feedstock to produce fuel ethanol. The ESE process was a true conservation measure whereas a material class which would have been discarded instead was the feedstock for the production of; Fuel Ethanol with a by-product of Conditioned Mash as Nitrogen Fertilizer. The application of the fertilizer from 2020 until it was stopped by EPA in 2021 covered approximately 2349.5 acres, totaling 27 fields.

The Site is located in Eastern Wichita County, Kansas, and is comprised of approximately 30 acres at the ESE Alcohol Facility and approximately 2349.5 acres of agricultural fields. ESE Alcohol used treated seed as a feedstock for ethanol production. The treated seed used by ESE Alcohol during the 2020 to 2021 time frame was treated with the following six (6) seed treatments: Clothianidin, Thiamethoxam, Tebuconazole, Thiabendazole, Iaconazole, Chlorantranilipole (the “Constituents of Concern” or “COCs”).

Two fields which received the higher applications of conditioned mash, most recently applied and one field which received the medium application rate will be chosen for the initial sampling event along with the facility and the conditioning area. These field types should indicate the highest potential to contain residual concentration of the seed treatments. This will also indicate which treatments should be analyzed going forward. Please see the attached listing (Table 1) showing the 20 pesticides the laboratory can analyze and as agreed to by EPA for the initial samples. Ten days prior to initial sampling activities the Project Manager will provide oral notification to the assigned EPA contact.

An Ecological Risk Assessment (ERA) was completed by EPA and included birds, mammals, and honeybees. Of the three assessment endpoints evaluated, honeybees were found to be the most sensitive assessment endpoint. The mechanism of exposure anticipated by EPA’s ERA is that pesticides are taken up by the roots of crops and translocated to the pollen of corn and nectar of certain plants none of which are cultivated in the area of concern, potentially exposing foraging bees to pesticide residue. Bee exposure is very unlikely due to the fact that

honeybees primarily seek nectar. Corn fields only have pollen and are not generally attractive to bees. It is important to keep in mind that any pollinator may be exposed in this manner; however, because honeybees are very sensitive to neonicotinoid pesticides, they are considered by the EPA to be a primary receptor of concern and remedial actions selected to be protective of honeybees should also be protective of other less sensitive pollinators.

The EPA ERA evaluated potential risk to honeybees by converting the maximum concentration in raw freshly produced WDG (although this material was not directly applied to fields) to a soil application rate in pounds of active ingredient per acre (lb a.i./A) using the arithmetic mean conditioned mash application rate of 7.04 tons/acre from 2020 through 2021. This estimate site-specific application rate was first compared to allowable maximum application rates for each pesticide to develop a list of seed treatments of Potential Concern. Clothianidin, thiamethoxam, ipconazole, tebuconazole, thiabendazole, and chlorantraniliprole. Site specific application rates of conditioned mash may have contained residue of these seed treatments which possibly exceeded the range of maximum label application rates identified by the Office of Pesticide Programs for these pesticides for crops typically grown in western Kansas.

## II. Sample Plan Procedure and Media to be sampled to determine duration of remediation crop production. (crop rotation limitation)

### A. Corn Fields-

The agricultural fields chosen for sampling as part of the initial round of sampling from Group 1 and Group 2 are identified in Table 3. These agricultural fields are used for production of wheat, corn, and/or milo, on a rotating basis in accordance with good agricultural practices.

Once the results from the initial samples with the highest potential to contain residual COC's have been received the determination for future sampling will be developed. In the event where COC, the list Pesticides in Table 1 are below the levels of ecological concern, i.e. maximum application rates or higher as may be established, no further sampling will be conducted on all agricultural fields in that Group level or lower Group level. In the event there are COC's above the levels of ecological concern a representative set of fields from the next tier of application levels will be sampled at the depth where COC's above levels of ecological concern were identified. The analysis will only be conducted for the COC's above the levels of ecological concern i.e. maximum label application rate. The sampling process will continue based on crop rotation requirements until the results indicate all COC's are below the levels of ecological concern. Once the field has an analysis of soil below the levels established that field will be removed from further sampling requirements.

### B. Field Soil Samples-

Soil sampling from representative agricultural fields will be undertaken to determine residual levels of COCs in the soil above maximum application rate for each pesticide, from land application of ethanol byproducts.

***Corn Fields to be Sampled.*** The conditioned mash application amounts varied based on the nitrogen fertilizer requirements at the time of application. The requirements were based on agronomic analysis of the soils in each corn field. For example the nitrogen fertilizer analysis, certain fields required 12 tons of mash to be applied while other fields only required three (3) tons of mash to be applied. 3 tons is the lowest application rate of conditioned mash utilized.

For purposes of the initial corn field soil sampling, the corn fields have been segregated into three (3) application groups based on historic application levels of mash: Group 1 - ten (10) tons per acre (tpa) and above, Group 2 – less than ten (10) tpi but greater than six (6) tpa, and Group 3 – less than six (6) tpa but greater than three (3) tpa. Within each group, the application date varies based on good farming practices.

Group 1 comprises ten (10) fields, totaling approximately 1075.5 acres. Group 2 comprises seven (7) fields, totaling approximately 489 acres. Group 3 comprises ten (10) fields, totaling approximately 785 acres for a total of 2349.5 acres.

For the initial round of field soil sampling, two (2) Group 1 fields and one (1) Group 2 field will be sampled. The soil samples will be analyzed for the COCs by LC/MS/MS at Matrix Laboratory N. W.

Due to the fact that Group 3 agriculture fields had a low application rate of conditioned mash, none of these will be sampled as part of the initial round of field sampling. Once sample results are received for the initial round of field sampling, we will review the analysis to determine whether Group 3 requires sampling; i.e. in the event that Group 1 and/or Group 2 COC levels are below maximum application rates of seed treatment, there will be no requirement for sampling the fields with the lower application rate of conditioned mash.

Fields chosen for sampling as part of the initial round of sampling from Group 1 and Group 2 are identified in Table 3.

**Sampling Procedures for Corn Fields.** Each morning and evening of sampling days, there will be a tailgate safety discussion among all parties. The primary safety items will be rubber gloves (change between each field soil sample) and safety glasses. The rubber gloves will ensure worker safety, as well as mitigate the possibility of cross-contamination between field soil samples. All supplies utilized for sampling will be new, clean and thoroughly decontaminated between fields.

Respondents will utilize a modified program of EPA's ISM recommendation. Respondents will utilize the Figures 2 and 3 provided as the basis of the sample protocol. Each circle pivot field in which conditioned mash was applied for Agronomic Value in a systematic approach will be considered. Two fields from application Group 1 and one field from application Group 2 will be selected based on criteria EPA provided; (1)

Highest application rate and (2) Recent Application. All of the conditioned mash application was completed no later than August 2021. Corn Field 1 (NW 32-16-17) in application Group 1 was selected due to the two applications of conditioned mash: February 2020 at 6.46 tons/acre and January 2021 at 10 tons/acre. Corn Field 2 (SW 32-16-37) in application Group 1 was selected due to the highest most recent application, approximately 12.2 tons/acre applied in January 2021. Corn Field 3 (SE 36-17-37) in application Group 2 was selected due to an application of 6.35 tons/acre in January 2020. (See Table 3) The two Group 1 fields are 92 acre circle pivot each. The Group 2 corn field is 160 acres. The three corn fields will be sampled in the same process for consistency.

**Sample Grid.** Each corn field will be placed into a 6 x 6 grid pattern overlay. The field edges whether circle pivot or square corn fields could have a slight variation as to the less mash applied due to stopping and restarting the spreader equipment. Therefore the field edges headlands will not be sampled to ensure sample aliquots are collected from the most uniform area of the corn fields. The circle pivot corn fields will be sampled in each complete grid (square portion) allowing for 20 cores, aliquots, near the center of each grid square. Each location will be selected and recorded via GPS.

(See Figure 2, Figure 3 and Map)

**Core Sampling.** Each core shall be 30" in depth divided in 10" segments. 0-10", 10"-20", 20"-30". Each core aliquots segment will be placed into a clean plastic 5 gallon bucket with a lid for mixing then sub-sampling. The large composite from 0-10" will be sub-sampled into 3 samples for analysis. One for EPA, one as primary, one as duplicate. The 0-8" composite shall serve as the third of upper depth sampling. The large composite from 10"-20" and 20"-30" depths will be sub-sampled into four samples, one for EPA, one for ESE, plus two replicates.

**Surface Sampling.** One additional aliquot will be collected from each grid square, approximately 100' from the core location. These 20 aliquots will be collected 0-8" then placed into a clean 5 gallon bucket with a lid for mixing then sub-sampling for one composite for EPA, and one composite for ESE. Once the aliquot collection is completed in one field the combined aliquots will be thoroughly mixed and sub-sampled.

**Sample Labeling & Shipment.** Once sub-samples are containerized, sealed and labeled they will be placed in a cooler for preservation until shipment via 2<sup>nd</sup> Day Delivery to the laboratory. Each composite sample will be labeled with the field, depth, name and date. Matrix Laboratory will analyze each sample via LC/MS/MS for the 20 analytes.

Once the results from the initial Group1 highest and Group 2 medium application samples are received the determination for future sampling will be developed. In the event there are COC's above the levels of ecological concern a representative set of fields from the next tier of application levels will be sampled at the depth where COC's above levels of ecological concern were identified. The analysis will only be conducted for the COC's above the levels of ecological concern in the 1<sup>st</sup> round of sampling results from Matrix Laboratory. The sampling process will continue after an established time interval i.e. when it is determined that corn may be planted back into the crop rotation after results indicate all COC's are below the levels of ecological concern i.e. maximum application rates. Once the field has an analysis of soil below the levels of ecological concern.

Three replicate samples will be submitted to the laboratory from each depth unit on each field in order to provide a measure of variability of the process.

**Decon Process**-Each and all items utilized in the sample collection process will be deconed upon the completion of sample collection in each field. All items will be wiped/brushed clean then rinsed with DI/Distilled Water three times to insure the removal of any residual material.

#### C. Staging and conditioning area-

Sampling from holding pond bottom material at the staging and conditioning area will be undertaken to determine residual levels of COCs in these materials, if any, for purposes of determining the appropriate disposition of these materials.

The staging and conditioning area (Figure 1) currently has approximately 2000 tons of pond bottom material on site in windrows. The pond bottom clean out material is the only ethanol production byproduct material remaining at the Site. This material was removed from the very bottom of the holding ponds where processed mash was stored prior to drying. The semi-dry mash was removed from the holding pond, transported to the staging and conditioning area for further conditioning and drying prior to application for its agronomic value as fertilizer and soil conditioner. The pond bottoms were then thoroughly cleaned and wheel compacted. The material was placed in windrows. The windrows are turned and rolled with a front end loader for further drying and conditioning. Each windrow will be sampled in seven random locations. Each sample location shall have two 4oz aliquots collected; one from 0"-6", then one from 12"-18", the next sample location will be sampled 0"-6" and 18"-24". This process will be repeated for a collection of 14 aliquots along each windrow. All fourteen 4oz aliquots from each windrow will be composited into a stainless steel container thoroughly mixed. Then 4 opposing sub-samples will be removed into sample container for analysis and to be split with EPA. Once containerized and properly labeled the sample will be placed in a secure container on ice. Once all samples are collected the batch will be shipped 2<sup>nd</sup> day to the laboratory for analysis.

#### D. Treated Seed Receiving Area

Soil sampling from the Treated Seed Receiving Area at the ESE Alcohol Facility will be undertaken to determine residual levels of COCs in the soil, if any. The level of concern at the facility shall be based on commercial/workplace levels for human/mammal exposure only. Treated seed used as a feedstock for ethanol production at the ESE Alcohol Facility was received at the Treated Seed Receiving Area depicted in Figure 6.

A composite soil sample will be obtained from the Treated Seed Receiving Area. One duplicate sample will be submitted from this area.

The area where the receiving grid was located will be the center of the sample zone. Eight sample locations will be sampled producing 8 aliquots (1)-5' and (1)-10' North of the receiving grid, (1)-5' and (1)-10' South of the receiving grid and (1)-5' and (1)-10' East of each previous sample point. One composite sample will be comprised of eight 4oz aliquots from 0"-6" all combined in a stainless steel vessel mixed thoroughly. Three opposing aliquots will be removed into sub-sample containers for analysis, one as a duplicate, and one split with EPA. Once containerized and properly labeled each sample shall be placed into a secure container with ice. Once all samples are collected the batch will be shipped 2<sup>nd</sup> day to the laboratory for analysis. The samples will analyzed by LC/MS/MS for the 20 analytes directed by EPA.

### III. Reporting

ESE Alcohol and Pioneer will submit sampling data from the sampling within thirty (30) days of receiving the data. The data shall be tabulated chronologically by media.

### IV. Schedule

Once the plan is accepted the sampling will be scheduled within two weeks, weather, crop condition, equipment and personnel permitting. The following schedule applies to the sampling work detailed in the sampling plan:

<b>Timeframe</b>	<b>Activity</b>
Within two weeks after approval of QAPP, DQOs, and HASP	Schedule Agricultural Field soil sampling, Staging and Conditioning Area, and Treated Seed Receiving Area Sampling. Sampling will not be scheduled 04-24-2023 thru 04-28-2023
10 days prior to sampling activities	Oral notification of sampling activities to EPA
Within 30 days after receipt of analytical results	Provide analytical results to EPA

## V. Sample Event Timing

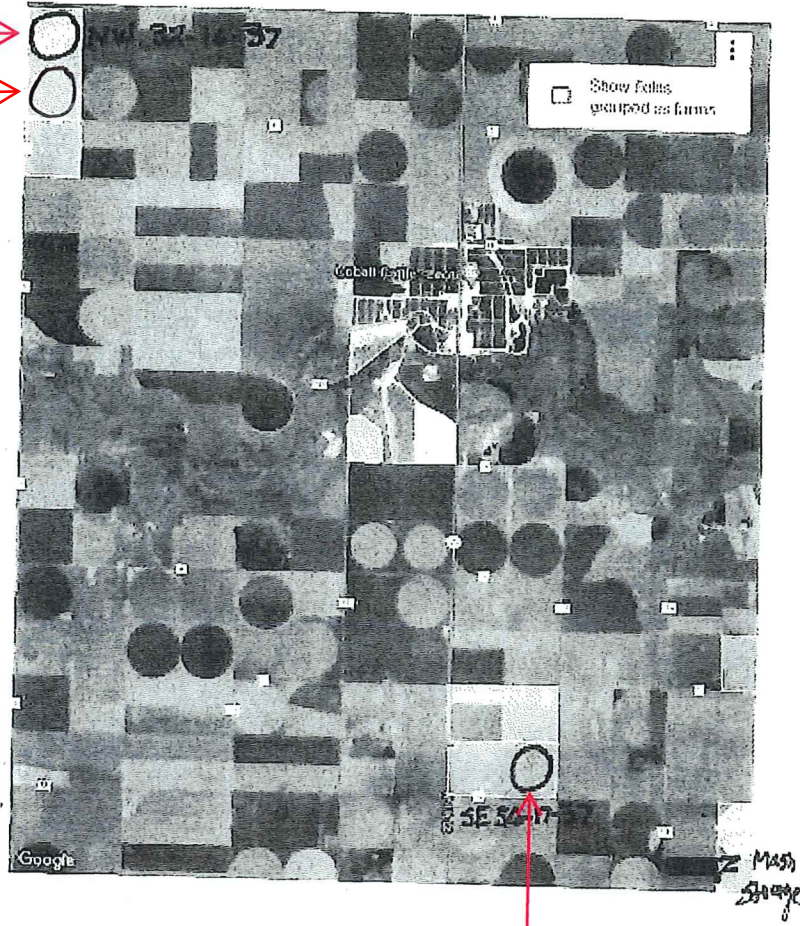
The timing of the next sampling event will be developed based on the results of the first sampling event, plus the timely crop rotation for planting of corn into the fields where conditioned mash was applied for the nitrogen fertilizer and soil conditioning. Once the results of the first sampling event are received and discussed subsequent action will be determined in conjunction with EPA.



# MAP

NW 32-16-37

SW 32-16-37



SE 36-17-37



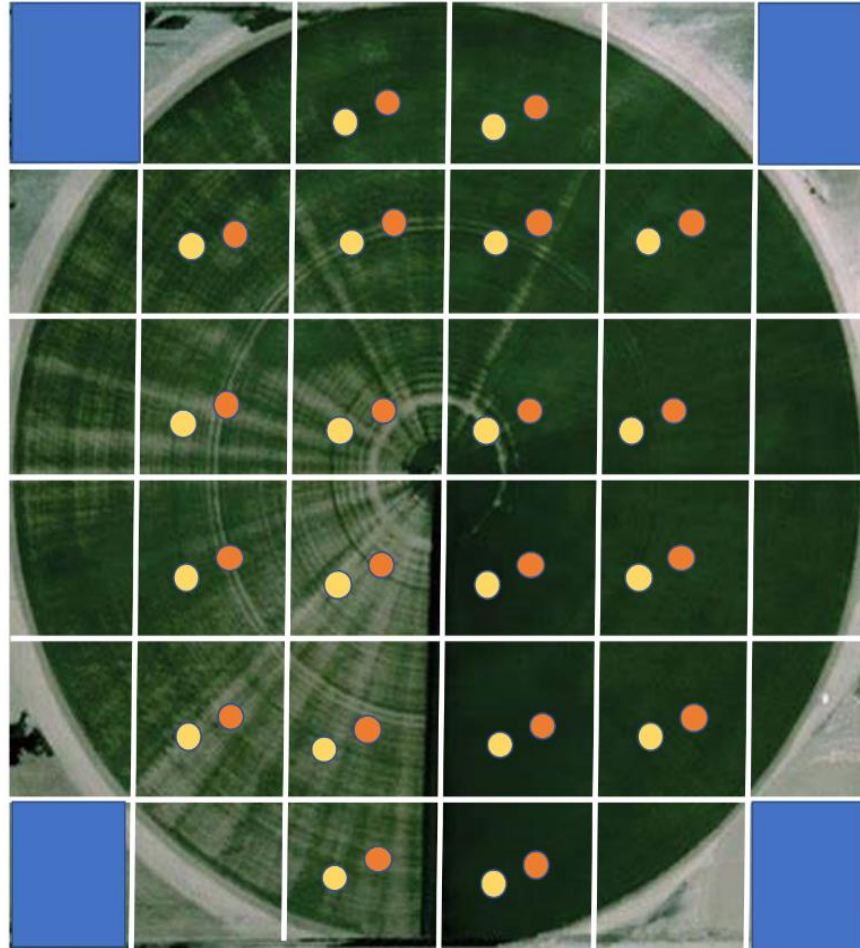
**FIGURE 2**  
**GROUP 1**

Sampling at 20 increments per Decision Units (Dus)

Group	# of Fields	# of acres	# of field to sample
1	9	794	2
2	5	451	1
3	7	786	0

**Objective:** Obtain 20 increments per DU per depth level

- Sample 6" surface and 2 centimeter (cm) soil core for random increments
- First surface increment is determined randomly, and subsequent increments are collected in the same relative location within each grid. This action is repeated for 20 soil core samples.
  - 20 surface increments at 0-6"
  - 20 soil core increments at 0-30"
- After collecting 20 surface increments and 20 soil cores, the soil cores will be subsampled and separated by depth.
  - 3 subsamples at surface"
  - 3 subsamples at 10-20"
  - 3 subsamples at 20-30"



---Internal Use---

**FIGURE 3**  
**GROUP 2**

Sampling at 20 increments per Decision Units (Dus)

Group	# of Fields	# of acres	# of field to sample
1	9	794	2
2	5	451	1
3	7	786	0

**Objective:** Obtain 20 increments per DU per depth level

- Sample 6" surface and 2 centimeter (cm) soil core for random increments
- First surface increment is determined randomly, and subsequent increments are collected in the same relative location within each grid. This action is repeated for 20 soil core samples.
  - 20 surface increments at 0-6"
  - 20 soil core increments at 0-30"
- After collecting 20 surface increments and 20 soil cores, the soil cores will be subsampled and separated by depth.
- Each set of 20 samples will be composited separately into a three main samples from which 3 representative subsamples will be sent for analysis.
  - 3 subsamples at surface"
  - 3 subsamples at 10-20"
  - 3 subsamples at 20-30"

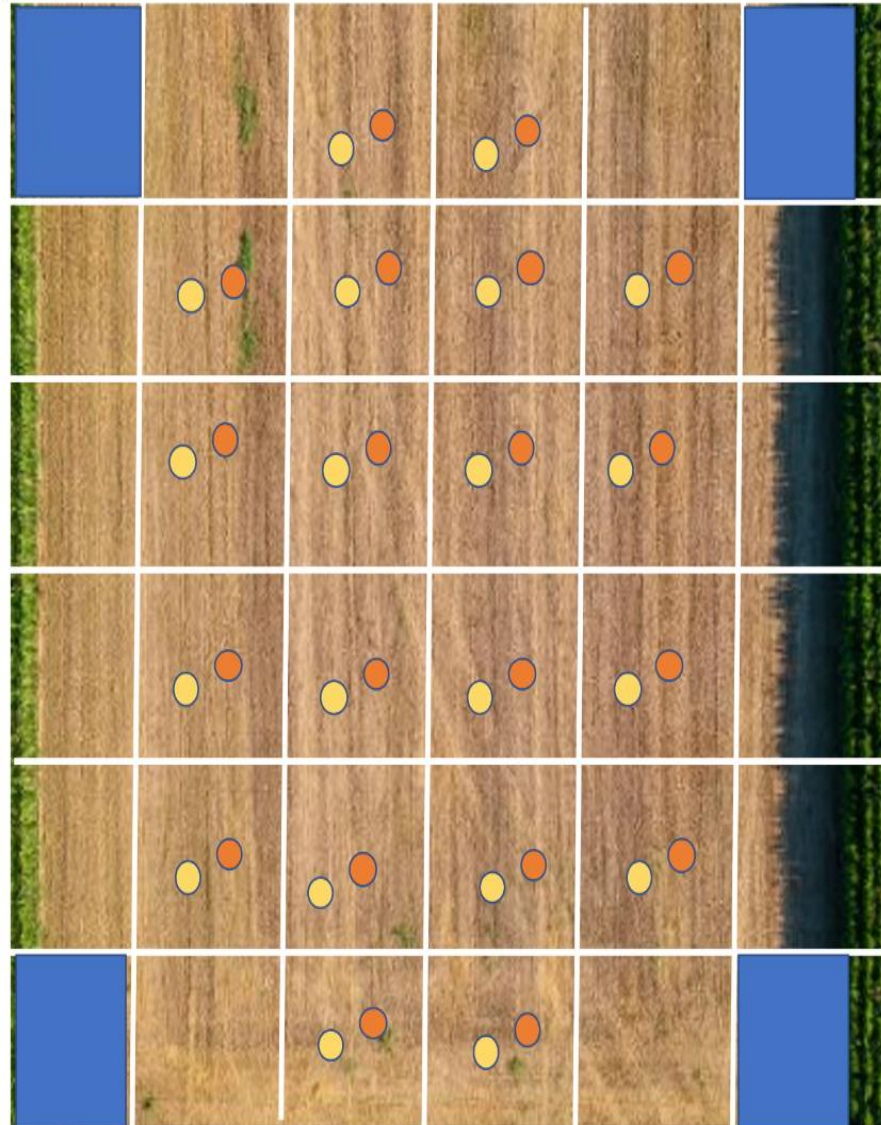
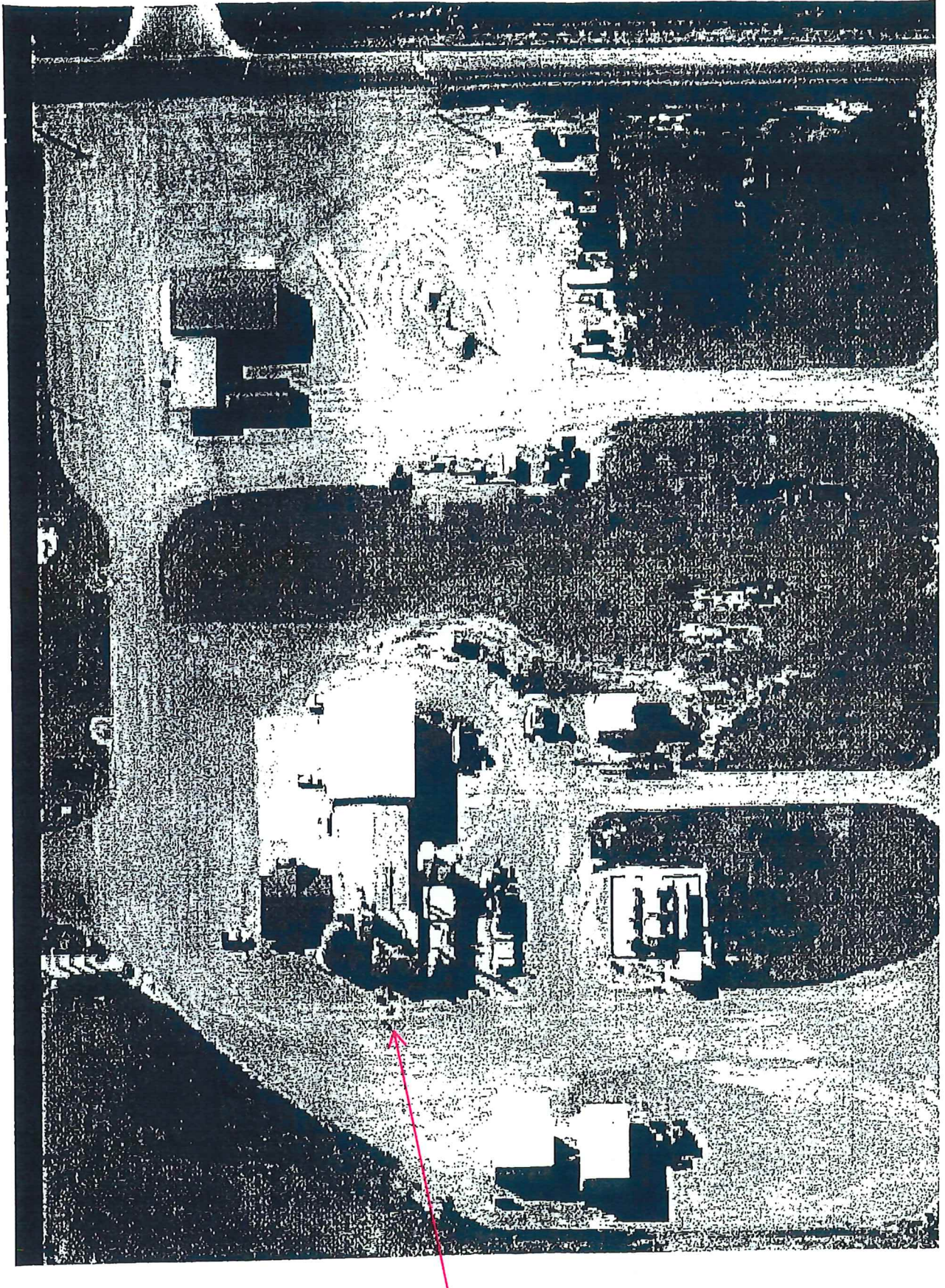




Figure 6

N →



Treated Seed Receiving Area



## Corn Figure

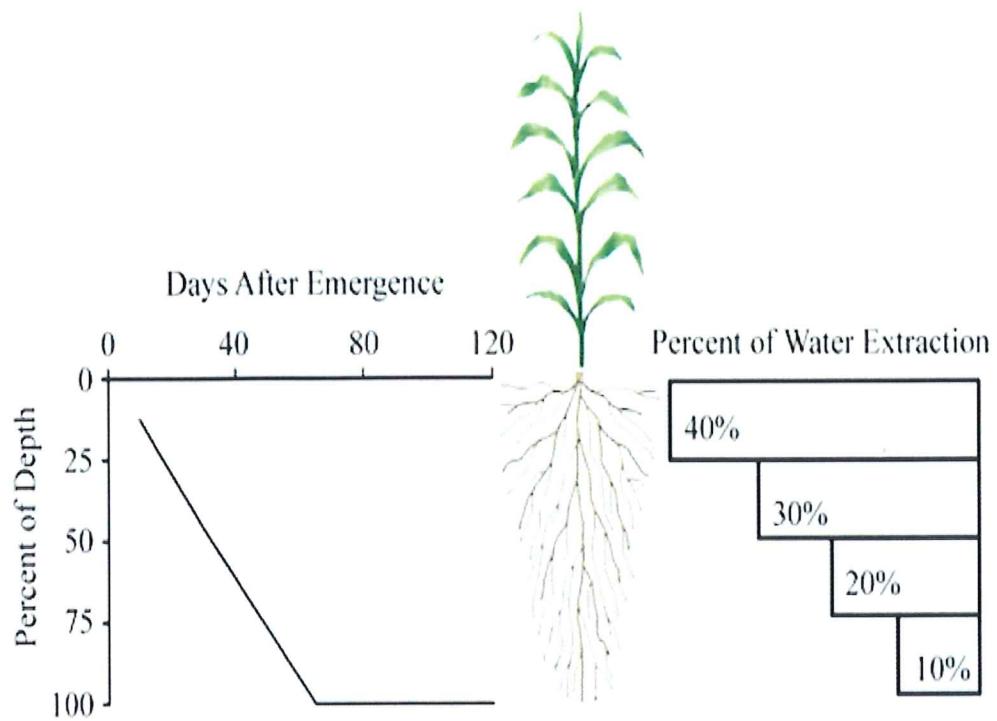


Figure 2. Depth of Corn Roots and Percentage of Water Extraction.

\*This diagram displays the uptake for corn to be 40% in the top 8" of the soil and 70% in the top 14"

Table 1

EPA Laboratory Analysis list of 20 Pesticides:  
For Initial Analysis

1. Azoxystrobin
2. Clothianidin
3. Chlorantraniliprole
4. Thiamethoxam
5. Thiabendazole
6. Mefenoxam
7. Ipconazole
8. Metalaxyl
9. Tebuconazole
10. Fludioxonil
11. Sedaxane
12. Abamectin
13. Captan ●
14. Carbendazim ●
15. Carboxin ●
16. Fluoxastrobin
17. Imidacloprid
18. Metconazole
19. Trifloxystrobin
20. Difenconazole

●EPA Removed

**Table 2**  
**Maximum Application Rate Chart**

Upon review of the maximum application rates listed in the chart provided by EPA 10 seed treatments were found to be below the published maximum application levels thus these rates were adjusted to the published levels.



<b>Pesticide</b>	<b>Maximum Application Rate (corn, wheat, soybeans) (lb/A)</b>	<b>Level of Concern (mg/kg)</b>	<b>Maximum Application Rate - References</b>
Azoxystrobin	5	2.5	Registration Review: Draft Risk Assessment of the Environmental Fate and Ecological Risk of Azoxystrobin
Clothianidin	0.2	0.1	Preliminary Bee Risk Assessment to Support the Registration Review of Clothianidin and Thiamethoxam
Chlorantraniliprole	0.2	0.1	Ecological Risk Assessment for Section 3 Registration for Fruit, Vegetable, Selected Field Crop, Turf and Ornamental Uses of Chlorantraniliprole (PC Code 090100)
Thiamethoxam	0.266	0.133	Syngenta Label
Thiabendazole corn 1.5lbs	1.5	0.75	Product Label
1oz/1000 sq ft			Problem Formulation for the Environmental Fate and Ecological Risk Assessment, and Endangered Species Assessments in support of Registration Review of Metalaxyl and Mefenoxam (Metalaxyl-M) - Product Label
Mefenoxam	2.72 lbs/acre	1.36	Mefenoxam (Metalaxyl-M) - Product Label
Ipconazole	7.43 lbs/acre	3.715	Ipconazole: Product Label
Metalaxyl	1.25	0.625	Problem Formulation for the Environmental Fate and Ecological Risk Assessment, and Endangered Species Assessments in support of Registration Review of Metalaxyl and Mefenoxam (Metalaxyl-M)
Tebuconazole	0.345	0.173	Ecological Risk Assessment for Section 3 Registration of Tebuconazole on Wheat, Cucurbits, Bananas, Turnips, Tree Nuts, Sunflowers, Soybeans, and Corn - Product Label
Fludioxonil	0.22	0.11	Registration Review: Preliminary Environmental Fate and Ecological Risk Assessment for Fludioxonil
Sedaxane	0.027	0.0135	Syngenta Product Label
Abamectin	0.061	0.0305	PRIA Label Amendment — Adding the New Use Crop Group 6 Product Name: Vibrance
Captan	4.48	2.24	EFED Registration Review Problem Formulation for Abamectin
			EPA Label Product 06-21-2019

Carbendazim	0.7	0.35	Thiophanate-methyl and MBC (Carbendazim): Draft Ecological Risk Assessment for Registration Review
Carboxin	0.4	0.2	Registration Review – Preliminary Problem Formulation for the Drinking Water and Ecological Risk Assessments of Carboxin and Oxycarboxin
Fluoxastrobin	0.5	0.25	Fluoxastrobin: Draft Ecological Risk Assessment for Registration Review
Imidacloprid	0.5	0.25	Imidacloprid -Transmittal of the Preliminary Terrestrial Risk Assessment to Support the Registration Review
			Metconazole 2 1 Metconazole: Draft Ecological Risk Assessment for Registration Review - Label
Trifloxystrobin	0.5	0.25	Bayer Crop Science Label Revised Ecological Risk Assessment for the Registration Review of Trifloxystrobin
Difenoconazole	0.13	0.065	Difenoconazole: Draft Ecological Risk Assessment for Registration Review

## Table 3

### Crop Fields

#### 1.) Circle Pivot Field NW 32-16-37

##### **Mash Applied**

Feb. 2020

Dec. 2020

##### **Rate**

6.46 Tons/Per Acre

12.0 Tons/Per Acre

##### **Crop Grown**

2020 Corn

2021 Corn

2022 Fallow

##### **Yield**

138 Bushels/Per Acre

216 Bushels/Per Acre

#### 2.) Circle Pivot Field SW 32-16-37

##### **Mash Applied**

Jan. 2021

##### **Rate**

12.2 Tons/Per Acre

##### **Crop Grown**

2020 Corn

2021 Corn

2022 Cover Crop

#### 3.) Circle Pivot Field SE 36-17-37

##### **Mash Applied**

Jan. 2020

##### **Rate**

6.35 Tons/Per Acre

##### **Crops Grown**

2020 Corn

2021 Corn

\*2022 Corn

##### **Yield**

156 Bushels/Per Acre

87 Bushels/Per Acre

109 Bushels/Per Acre

\*Purchased Commercial Fertilizer Due To Lack of Mash To Apply

## **Application Equipment**

The historical application of the conditioned mash by-product for its agronomic value is via a manure spreader controlled by GPS guidance. The equipment for the application of the conditioned mash by-product is very similar to the equipment utilized to apply chicken litter for the fertilizer value. These fields are no-till therefore the mash by-product is not incorporated into the soil. The rate of application varies greatly between fields based upon the agronomic requirements of each field based on agricultural analysis. The application is consistent within each field area based on nitrogen fertilizer requirements.